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(19)

(54) METHOD FOR SEALING LEAKAGE POINTS IN GAS PIPES

(71) We, SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ N.V., a company organized under the laws of the Netherlands, of 30, Carel van Bylandtlaan, The Hague, the Netherlands, do hereby declaire the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to sealing leakage points in pipes; more particularly it relates to sealing of mechanical joints in gas mains.

Mechanical joints contain a rubber gland, but there is still a considerable space between the elements of the joint on the gas side of the pipe. Packed joints are of another type. They contain fibrous materials as a packing and there is no considerable space between the elements of the joint on the gas side of the pipe.

When natural gas is transported through gas mains having joints, after some time these joints may leak due to extraction by the dry natural gas of relatively volatile components of the packing. This type of leakage is especially encountered when existing gas mains are changed over from transporting of relatively wet artificial gas such as coal gas to transporting the dry natural gas.

The problem of sealing leaking points in pipes and in particular leaking joints in gas mains is not simple. The application of electrophores to deposit sealing material is not successful because the sealing material deposits only on the conducting walls of the pipes and not on packings which are non-conducting. British patent specification 1,062,278 describes the use of bitumen for sealing leaks, the method described therein comprising treating a pipe with an aqueous emulsion comprising a bitumen and an emulsifier. In packed joints, the emulsion penetrates into the packing and breaks, leaving bitumen in the pores and interstices of the packing, thereby successfully sealing the pores and interstices against the passage of gas. The rheology of the bitumens described in Specification 1,062,278 does not favour their retention in the gap of a mechanical joint. These bitumens are mainly viscous in behaviour and when the emulsion is withdrawn from the pipe they tend to drain away (as well as the emulsion) and, in that case, are lost.

According to the present invention a method of sealing a leakage point in a pipe, comprises treating the pipe with an aqueous emulsion comprising a blown bitumen and an emulsifier.

The invention predicated on the use of a material having gelatinous properties. Blown bitumen does not behave as a viscous liquid, such as the bitumens described in Specification 1,062,278. Blown bitumens are gelatinous and when an emulsion containing a blown bitumen penetrates into the leakage points of the mechanical joint and breaks, blown bitumen is deposited in the gaps. The deposit formed cannot run down under its own weight due to its gelatinous properties. Of course, these emulsions containing blown bitumen can also be used to remedy leakage points in packed joints containing fibrous material as a packing.

Preferably, the emulsion is formulated such as to obtain an emulsion having gelatinous properties. For this purpose, use can be made of gelling agents, such as hydroxyalkyl methyl cellulose (e.g. hydroxypropyl methyl cellulose sold under the brand name "Methofas"), carboxymethyl cellulose, glue, lecithin, polyvinyl alcohol and other compounds known for their gelling properties. These preferred emulsions, which preferably contain 0.1—5%w of gelling agent, are also effective in sealing leakage points in case the emulsion does not break quickly.

Shrinkage of the rubber in mechanical joints is a frequent cause of leakage. This may effectively be remedied by using the emulsions according to the invention which upon breaking deposit blown bitumen onto or in the pores caused by the shrinkage. The presence in the emulsion of a mineral oil or a mineral

oil component, preferably one having a low volatility,is of further advantage for sealing leakage points caused by shrinkage of the rubber packing since rubber absorbs mineral oil or components thereof and this causes the package to swell.

In practising the sealing of leakage points according to the invention, use can be made of the mode described in British patent specification 1,062,278. A section of pipe can be isolated and completely filled with emulsion, whereby emulsion enters a leakage point; thereafter the pipe is emptied of the emulsion. It is to be understood that in certain circumstances it may not be necessary to completely fill the pipe. The filling can be done by a pump or by means of a suitable head of pressure, as in the case of a container on the ground connected to a town gasmain about 3 metres underground. During the filling, gas will be vented from the pipe. The pressure of the emulsion in the pipe and the time the emulsion is in the pipe (including at least part of the time for emptying the pipe of the emulsion) will contribute to the effectiveness of the seal. Breaking of the emulsion in the leakage point may occur when the emulsion enters the leakage point or it can be produced afterwards, for example, by passing air through the isolated section of the pipe after the emulsion has been emptied out. In the latter case, instead of passing air through the gas main, the supply of gas could be restored to produce the breaking of the emulsion if the gas is sufficiently dry.

The emulsion,preferably, comprises 20 to 70% by weight of dispersed material (i.e. the bitumen-containing phase). At the upper end of this range, the emulsion can be regarded as a concentrate for dilution. However, whether it is diluted or not before use may be dictated by practical conditions or may be a matter of convenience.

As explained above, a blown bitumen is used, for example, a blown bitumen having a softening point Ring and Ball of 50—140°C and a penetration at 25°C of 3—60 dmm. Examples are blown propane bitumen having a softening point of 50—60°C and a penetration of 3—10 dmm and blown straight-run bitumen having a softening point of 110—120°C and a penetration of 10—20 dmm. Preferably, a blown bitumen is used having a softening point of 90—100°C and a penetration at 25°C of 20—30 dmm. The known R 85/25 grade is such a preferred bitumen, for example, Mexphalte R 85/25 (Mexphalte is a Registered Trade Mark). Mixtures of blown bitumens can be used to make the emulsion. When low-volatile oil components are added an emulsion is obtained containing a swelling agent for rubber packing as set out hereinbefore. Suitable products containing low-volatile components are heavy aromatic extracts obtained from lubricatng oils, such as Dutrex grades (Dutrex is a Registered Trade Mark), for example, Dutrex 217 having an aromatics content of 83%wt. If desired, these aromatic extracts of lubricating oils, which show a relatively high viscosity, may be diluted with lower molecular weight aromatics, for example, aromatic solvents, preferably, aromatic white spirit having a boiling range of 165—185°C and an aromatics content of 80%wt or above.

Suitable blends of blown bitumen and an aromatic extract are given in Table I.

TABLE I

No.	Composition		Penetration dmm at 25°C of the composition
1.	blown propane bitumen 95/5 aromatic luboil extract (Dutrex 217)	90%w 10%w	15
2.	blown propane bitumen 95/5 aromatic luboil extract as in composition 1	80%w 20%w	20
3.	blown bitumen 85/25 aromatic luboil extract as in composition 1	90%w 10%w	29
4.	blown bitumen 85/25 aromatic luboil extract as in composition 1	95%w 5%w	47

Compositions as shown in Table I may be diluted with aromatic solvents to increase the penetration, if desired. For example, composition 4 of Table I may be diluted with respectively 5°w, 7%w and 9%w (calculated on the blends obtained) of aromatic white spirit (boiling range 165—185°C, 80%w aromatics) to obtain blends having a penetration (dmm) at 25°C of 96, 135 and 203, respectively. These blends can be easily emul-

sified with water and an emulsifier to obtain emulsions suitable for the method of the invention.

The emulsifier used for emulsifying blown bitumen can be any suitable emulsifier or mixture of emulsifiers and as a rule can be present as 0.2 to 5% by weight on the aqueous phase of the emulsion. Suitable emulsifiers include anionic emulsifiers (for example, alkali metal salts of organic acids, for instance, the potassium salt of wood rosin acid, which acid is obtainable by destructive distillation of pine wood chips and sold under the Registered Trade Mark Vinsol, and a 50/50 mixture of the said potassium salt and lactic casein) cationic emulsifiers (for example, salts of fatty amines, for instance, a mixture of alkyl-amine propylamines containing from 16 to 18 carbon atoms in the alkyl group and sold under the name Duomeen T (the word "Duomeen" is a Registered Trade Mark), or salts of fatty amides or fatty amino acids, proteins (for example, lactic casein) and colloidal clays (for example, bentonite). A typical example of a suitable emulsion is one obtained by emulsifying 64.3 parts by weight of a blend of 86%w blown straight-run bitumen R 85/25, 5%w aromatic luboil extract and 9%w of aromatic white spirit (boiling range 165—185°C, 80%w aromatics) and 35.7 parts by weight of aqueous phase containing an emulsifier comprising a diamine (C_{16}—C_{18}-alkylamino-propylamine), imidazoline emulsifier (made by condensing diethylene triamine with fish-oil fatty acids) and Normal aqueous hydrogen chloride in a quantity calculated on the aqueous phase of 1.2%w diamine, 0.25%w imidazoline emulsifier and 0.62%w Normal aqueous hydrogen chloride. The viscosity at 20°C of this emulsion is 21.7°E.

As set out hereinbefore, emulsions having gelatinous properties are the preferred emulsions for carrying out the method of the invention. Such an emulsion has, for example, the following composition:

blown bitumen composition	60 parts by weight	100 parts by weight
aqueous phase	40 parts by weight	
hydroxypropylmethyl cellulose	0.15 parts by weight,	

wherein the blown bitumen composition is a mixture of 86%w blown straight-run bitumen R 85/25, 5%w aromatic luboil extract (Dutrex 217) and 9%w aromatic white spirit (boiling range 165—185°C, 80%wt aromatics) and wherein the aqueous phase contains 1.2%w C_{16-18}-alkylaminopropylamine, 0.25%w imidazoline emulsifier and 0.62%w Normal aqueous hydrogen chloride, the balance being water. Such an emulsion has the following properties:

retained on a 0.74 mm sieve, %w	less than 0.1
viscosity °E at 20°C	6.9
cS at 20°C	52

When in the above emulsions the quantity of hydroxylpropylmethyl cellulose (Methofas) on emulsion is increased to 0.5%w or to 2.0%w, the viscosity increases as may be seen below:

	0.5%w Methofas	2.0%w Methofas
viscosity °E at 20°C	16.6	—
cS at 20°C	126	—
viscosity, Ferranti at 25°C, cP		
at 13 sec.$^{-1}$	—	1090
25	186	646
37	165	—
54	—	440
104	106	—
258	88	—

Another example of a gelling agent is a carboxymethyl cellulose, for instance, sodium carboxymethyl cellulose. Suitable concentrations are 0.5—1.0%w calculated on the aqueous phase of the emulsion. The viscosity of an emulsion as specified above wherein Methofas was replaced by 0.5%w sodium carboxymethyl cellulose as measured in the Ferranti viscometer is as follows:

viscosity, cP	
at 21 sec.$^{-1}$	260
at 106 sec.$^{-1}$	79

EXAMPLE

The method of the invention, using various emulsions comprising a blown bitumen, was tested in a test rig simulating a short length of a gas main. The test rig comprised a short length of 3 in cast iron pipe sealed at either end. The pipe had a bell and spigot joint between its ends in which there was a packing consisting of 40 cm of wet jute rope. A white metal collar was provided for compressing the packing to a controlled degree to give a degree of leakage in the joint convenient for test purposes. The compression of the packing was produced by drawing the metal collar into the joint by screws.

In all the tests given below the jute packing was compressed to a density of 0.6 g/cm^2. At this density the rate at which air leaked from the rig at a pressure of 50 millibar was between 5 and 8 litres/minute.

The emulsion was tested by filling it into the rig and maintaining it at a pressure of 1 bar for 100 minutes. The emulsion was then drained out and the rig ventilated by passing air through it at 1 litre/minute at 50 mb for 500 hours. At intervals the rig and its manometer were isolated and the pressure decay measured over 5 minutes. At the end of the ventilation period the test pressure was increased to 1 bar for one hour and the leak rate measured again.

Test No.	Disperse phase	Continuous phase
1	86%w R85/25 bitumen 5%w Aromatic oil 9%w Aromatic solvent	1.2%w Duomeen T 0.25%w Imidazoline emulsifier 0.62%w Hydrogen chloride →100%w Water
2	As test 1	1.2%w Duomeen T 0.25%w Imidazoline emulsifier 2.0%w Methofas PM 450 0.62%w Hydrogen chloride →100%w Water
3	As test 1	1.1%w Duomeen T 0.5%w Triethanolamine 0.5%w Nonidet P40 5.0%w Diethylene glycol 0.62%w Hydrogen chloride →100%w Water

When these compositions were tested in the manner described the leak rate at 50 mb and 1 bar was in each case below the limit of detection by the method used; that is, less than 0.1% of the original leak rate.

WHAT WE CLAIM IS:—

1. A method of sealing a leakage point in a pipe, which method comprises treating the pipe with an aqueous emulsion comprising a blown bitumen and an emulsifier.

2. A method as claimed in claim 1, in which a pipe is filled with the aqueous emulsion and then emptied.

3. A method as claimed in claims 1 and 2, in which the emulsion comprises 20—70% by weight of a blown bitumen having a softening point Ring and Ball of 50—140°C and a penetration at 25°C of 3—60 dmm.

4. A method as claimed in claim 3, wherein the blown bitumen has a softening point of 90—100°C and a peneration at 25°C of 20—30 dmm.

5. A method as claimed in claims 1—4, wherein the emulsion comprises an aromatic extract of a lubricating oil.

6. A method as claimed in claim 5, wherein the emulsion comprises an aromatic white spirit.

7. A method as claimed in claims 1—6, in which the aqueous phase of the emulsion comprises 0.2—5% by weight of an emulsifier.

8. A method as claimed in claim 7, in which the emulsifier comprises a C_{16}—C_{18}-alkylaminopropylamine salt.

9. A method as claimed in claims 1—8, in which the emulsion contains a gelling agent.

10. A method as claimed in claim 9, in which the gelling agent is chosen from the cellulose, a carboxymethyl cellulose, glue, lecithin and polyvinyl alcohol.

11. A method as claimed in claims 9 and 10, in which the emulsion comprises 0.1—5% by weight of gelling agent.

12. A method of sealing a leakage point in a pipe, substantially as described.

13. A method of sealing a leakage point in a pipe, substantially as described in the Example.

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